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**PORTABLE ELECTRONICALLY BASED METHOD FOR THE
NONDESTRUCTIVE REMOTE SENSING OF CREVICE
CORROSION**

Contract No.: N00014-93-C-0067

FINAL TECHNICAL REPORT

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1. INTRODUCTION

A. OBJECTIVE

The objective of this SBIR Phase I investigation was to demonstrate the feasibility of detecting localized crevice corrosion of gasketed flanges in pipeline joints by four-point probe resistance measurements. The specific aims were: (i) to evaluate the accuracy of the four-point probe resistance method in detecting changes in the thickness of model cavities produced in metal specimens, using DC and/or AC modes; (ii) to investigate the detection limits of artificially created pits on different types of alloys, their thicknesses and shapes, using the best method determined in specific aim (i); and (iii) to apply the procedures evaluated in specific aims (i) and (ii) to locate crevices of different sizes formed in a metal specimen whose geometry approaches that of a flange.

B. BACKGROUND

One of the most dangerous and hard-to-detect type of corrosion occurring in systems containing joints, like pipelines, is crevice corrosion. Crevice corrosion occurs in narrow gaps, e.g., if a flange and a gasketed joint lose their tightness. In that case the solution entering the crevice, which might be open or blind, causes localized corrosion to initiate leading in most cases to pit formation. Pit formation¹ is affected by factors such as the solution composition, various properties of the metal and by the crevice geometry^{2,3,4,5}. Among the ions present, Cl^- ions play a decisive role in the initiation of pitting by breaking through the passive oxide film present on the surface of the metal. This is especially pronounced where sea-water flows through pipelines.

1. Crevice Corrosion Of Gasketed Joints

Corrosion in the crevices of gasketed joints in the presence of sea-water has been particularly troublesome for the Navy in assembled sea-water pipe networks. The disassembling of numerous pipe joints, e.g., gasketed flanges, is a tedious, slow and costly way to inspect for crevice corrosion. Therefore, a need exists to search for new nondestructive techniques for crevice corrosion detection. Some of the existing non-destructive techniques, e.g., electrochemical techniques: scanning current probe and scanning potential probe, are based on corrosion examination from the water side, i.e., by introducing sensors inside pipelines. These are slow, impractical and not necessarily relevant to localized crevice corrosion. There are techniques which can be used without entering the pipeline system, like ultrasonics, radiography, eddy current measurements and neutron radiographic spectroscopy. However, most of them suffer from low signal-to-noise ratios due to spurious signals caused by scattering on rough-pitted surfaces.

The innovative approach investigated here is based on the application of four-point probe resistance measurements for the detection of pit formation and thickness changes in gasketed pipe joints caused by crevice corrosion. This approach is a non-destructive method and very simple to develop. The main advantages are that the sensing probe apparatus can be easily adapted for inspection of different sizes of flanges, quickly mounted and dismantled, easily maneuvered on the outer side of the joint, and the costs for development and application of this technique will be very low. Compared with other non-destructive techniques, four-point probe resistance measurements, applied from the outer side of the flange has the capability of detecting local changes in thickness, due to crevice corrosion, taking place at the inner (water) side of the flange. Thus, a whole flange material can be thoroughly inspected and an image of the corrosion damage created.

2. Detection of Gasketed Flange Crevice Corrosion by Four-Point Probe Resistance Technique

The detection of crevice corrosion in gasketed flanges, or generally in other components of pipeline networks subjected to localized corrosion, is based on measurements of changes in resistance of the flange alloy caused by localized changes in thickness due to crevice corrosion. The probe is attached on the outer surface of the flange, and there is no need for disassembling the flange. By multiple and repeated resistance measurements of the whole area of possible attack by crevice corrosion, and after graphical data processing by a computer, an image of the flange damages can be obtained.

Metallurgical variables have an affect on the resistance since they disrupt the crystalline structure. The main factors influencing resistance are: temperature, alloys and phases, impurities, and thermal treatments. The influence of temperature is generally linear for metals and it increases resistance approximately 0.4% per degree Celsius. This is not a serious limitation of the proposed method, because measurements are made by a current pulsing technique which does not induce significant temperature changes in a tested specimen. Thermal treatments may affect the resistance by two mechanisms. The first mechanism involves modifying the phase equilibrium. For example, quenched steel has a martensitic structure, while annealed steel is a combination of ferrite and pearlite. The second mechanism involves the phenomenon of second phase precipitation. Precipitation hardening occurs in many alloys such as aluminum, beryllium, copper and in some stainless steels. In the annealed state these alloys attain their minimum resistance while in their treated state they attain maximum resistance. The resistance of these alloys is a good indication of the metallurgical state and therefore of the mechanical properties. Thus, resistance measurements may be used to determine the carbon content and thermal treatment of stainless steel alloys. As a non-destructive method, resistance measurements have been used to evaluate spot welds. There is a good correlation between the mechanical properties of the spot weld and the potential drop. The microresistance is proportional to the fused surface, the larger the fusion area, the smaller the resistance.

The most common method for measuring resistivity is the four-point probe method and was used throughout this Phase I study. A known current, I , is applied between the two outer probes while the potential difference, V , is measured between the two inside probes. Because no current is flowing in the voltage circuit, contact resistance of the potential probes does not affect the voltage measurements. Thus, errors created by cable and contact resistances are eliminated using the four-point probe method.

An analytical solution for the uniform resistivity layer over an insulating boundary is^{6,7}:

$$R(W,s) = (\rho/\pi W) \ln \{ \sinh (W/s)/\sinh (W/2s) \} \quad (1)$$

and

$$R(x,s) = (\rho/\pi W) \ln \{ \cosh (W/s)/\cosh (W/2s) \} \quad (2)$$

for a conducting boundary. In both cases the probe spacing, s , and the distance, W , to the boundary appear as the ratio W/s . This ratio determines the behavior of \sinh and \cosh functions. For the situations where the distance to the boundary is small compared to the probe spacing, i.e., $W/s < 1$, the \sinh function may be replaced by its argument whereas the \cosh function approaches unity. Values for different W/s ratios, can be found tabulated in the literature in terms of a correction factor, CF^8 . According to Van der Pauw⁹, if probes are equispaced a distance, s , apart, resistivity, ρ , is given by:

$$\rho = \frac{RW \pi}{\ln 2} \quad (3)$$

where $\pi/\ln 2$ is the geometrical correction factor ($CF = 4.5$)¹⁰. This equation is valid for cases where $W < s/2$, and it shows that the resistivity depends linearly on the thickness of the specimen. If $s/2 < W < 3s$ then the correction factor has to be taken into account:

$$\rho = \frac{RW \pi}{\ln 2 CF} \quad (4)$$

When the thickness of the specimen is greater than three times the electrode spacing ($W > 3s$) the resistivity is a function of spacing, s :

$$\rho = 2\pi s R \quad (5)$$

Equations (4) and (5) show that the thickness of thick specimens can be measured by adjusting the spacing between the four point probe contacts. The resistivity probe is sensitive to the thickness where $W < 3s$.

When the dimensions of the specimen are smaller than the probe spacing, i.e., in length and width, resistivity measurements are affected by edge effects. The resistivity data can then be corrected by multiplying by geometric correction factors, e.g., for rectangular and/or circular geometries. For simple geometric shapes correction factors for the edge effects can be found tabulated in the literature⁸.

II. EXPERIMENTAL PROCEDURES AND METHODS

A. ALLOY SAMPLES

Several types of metal samples were used: aluminum plates, carbon (SAE 4140) and stainless steel (SS 316) foils, discs and rings. Carbon steel flanges (Weldbend, Inc.), designed for 1.5" diameter and 150 psi pressure pipelines, were also tested. Although there are several new categories of highly corrosion-resistant stainless steels for use in sea-water and sea-water cooled systems (for a Review see Ref¹¹), most of the work performed concentrated on Inconel alloy 625, the material proposed by the Navy. Inconel alloy 625 has a typical chemical composition of: 58% Ni, 20-23% Cr, 5% Fe, 8-10% Mo, 3-4% Co or Ta and ca 0.1 % carbon. Plates, of 12"x12" area and 0.5" thick, were purchased from Inco Alloys International, Inc, Huntington. Alloy samples were not specially pretreated before resistance measurements, except for degreasing with acetone and distilled water.

Crevice (cavities) of different sizes and depths were machined in the metal samples used. Critical dimensions (e.g., thickness) of samples and crevices were determined using digital micrometers and calipers (Mittutoyo) with an accuracy of 0.01 mm or better.

B. RESISTIVITY PROBES

One of the main goals of this Project was to design a four-point test probe which would assure an accurate and repeatable way of contacting the metal surface under test. The spacing between probe contacts is an important factor in determining the thickness of test materials. Both commercial and in-house fabricated four-point resistance probes, with different spacings between contacts and different contact spring pressures, were used

throughout the study. An Alessi standard contact probe station, model CPS-05 provided pressure controlled contacts of either 40 - 70 g per tip or 70 - 180 g per tip, when used with C4S-475S and C4S-645S probe heads, respectively. Low contact resistance was achieved by using osmium tips of 0.005" radii. Spacing between the tips in these commercial probe heads was 1 and 1.5 mm, respectively. Ultraoptec, Inc. resistivity probes, with spacing between the contacts of 1.5 and 9 mm, respectively, were also used. In order to be able to measure large and thick specimens, probe heads allowing larger spacing between the tips and a special circular (curved) arrangement of the contacts, were designed in-house. Figure 1 shows one of the typical designs of four-point probes with spacing of 1.0 cm between the spring loaded contacts (Newark, S3B4G). Contacts were 30° spear point, gold-plated berillium-copper, designed to pass 5 A continuous current, with 6.35 mm full stroke and spring force of 59 - 113 g.

In all the experiments carried out in this study, changes in resistance due to the presence of artificially made crevices of different depths were measured by placing the four-point probe at the side opposite to where the crevices were made. Thus, a non-destructive mode of detection of crevice corrosion of gasketed materials was assured.

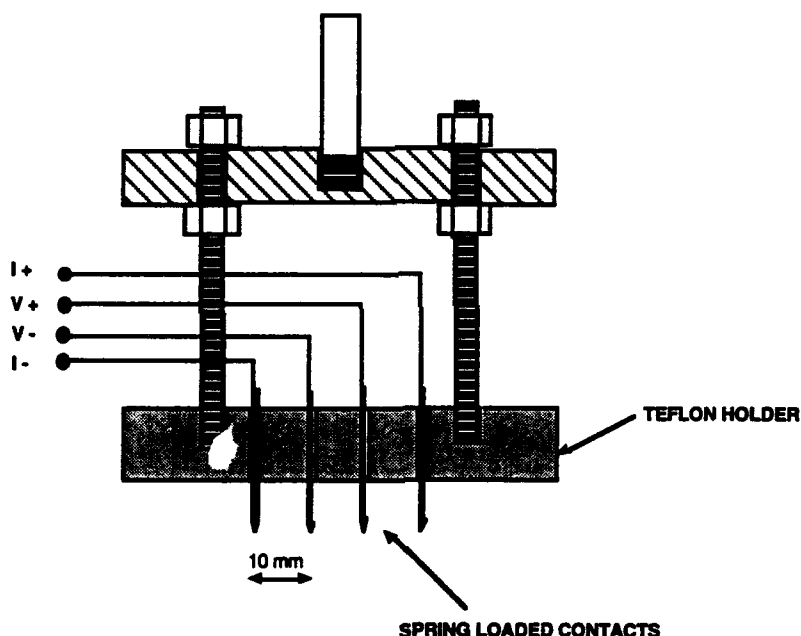


Figure 1. Schematic of a four-point probe head designed for resistance measurements and thickness determinations of thick metal plates.

C. INSTRUMENTATION

Four-point probe low resistance DC measurements were performed using a Valhalla Scientific Digital Micro-Ohmmeter, Model 4300B. The Model 4300B provides six ranges of user selectable test currents (from 0.1 mA to 10 A) and three voltage sensitivity settings (20 mV, 200 mV and 2 V). The unit's 4 1/2 digit resistance reading allows basic accuracy of $\pm 0.03\%$. Although the instrument possesses a temperature sensor for room temperature compensation, it does not compensate for possible local overheating effects, especially when high DC currents are passed. For 10 A DC current applied, a resolution of $0.1\ \mu\Omega$ is specified. Pulsed current resistance measurements were performed using an Ultraoptec, Inc. digital microvoltmeter with an interface board for real-time computer

measurements, yielding a sensitivity of $0.01 \mu\Omega$. The resistance was measured by application of short (few milli-seconds) current pulses of 0.5 or 5 A amplitude at the two outer contacts of the four-point probe, and by sensing the voltage at the two inner contacts. Compared to the DC technique, where continuous current is applied, this technique has the advantage of not overheating the sample at the electrical contact points, hence, avoiding temperature effects on micro-resistance measurements.

III. RESULTS AND DISCUSSION

A. TESTING EXPERIMENTAL ARRANGEMENT FOR RESISTIVITY MEASUREMENTS

The experimental setup for resistivity measurements using a pulsed current technique was tested by measuring the resistivity of SAE 4140 steel discs, of 9.65 cm diameter. A commercial Alessi 645 S probe, with osmium tips of 0.13 mm radii, 1.57 mm spacing and 70-180 g spring pressure was used. Table 1 summarizes the results. For the Alessi probe the W/s ratio was higher than 3 and Equation (5), which is valid when resistance depends on probe spacing was used in calculating resistivities. Resistivity values obtained for three specimen thicknesses were reproducible and corresponded to the known resistivity value of SAE 4140 carbon steel. SAE 4140 possesses a resistivity of ca, $23 \mu\Omega$ cm in the annealed state and $32 \mu\Omega$ cm in the quenched state. Data obtained indicated that the steel samples used were annealed.

Table 1. Resistivity measurements performed on SAE 4140 carbon steel discs using a current pulsing technique.

4 POINT PROBE	PROBE SPACING S/cm	SAMPLE THICKNESS W/cm	W/S	RESISTANCE $\mu\Omega$	RESISTIVITY $\mu\Omega$ cm	Equation used for calculations
Alessi 645S	0.15	1.385	12.23	20.60	19.4	(5)
		1.912	12.75	21.68	20.4	(5)
		2.619	17.46	21.52	20.3	(5)

In order to consistently apply this technique for the detection of crevice corrosion of gasketed flanges, probes should be designed with the spacing between the tips corresponding to the ratio $W/s < 3$, i.e., a condition where resistivity is dependent on the thickness of a corroded material. Figure 2 shows the dependence of the resistance of stainless steel (SS 316) plates (16 x 30 cm) on the thickness of the samples. Four different probes, commercial Alessi and home-made probes with gold-plated contacts, were used with contact spacings of 0.15, 0.5, 1.0 and 1.5 cm, respectively. On designing the probe spacing such that the ratio $W/s < 3$, the resistance was dependent on the thickness of the sample. This is clearly demonstrated in Figure 2.

B. COMPARISON BETWEEN RESULTS DERIVED FROM CONSTANT CURRENT AND PULSED CURRENT TECHNIQUES

Resistance values for relatively thick carbon steel discs (SAE 4140) using a constant DC current technique and a current pulsing technique are compared in Table 2. The current pulsing technique allowed almost two orders of magnitude higher sensitivity

and showed better reproducibility than the constant DC technique. Although the four-point probe resistance is theoretically independent of the probe current density and the probe radius¹², DC resistance measurements exhibited small differences in resistance when 1A and/or 10A constant currents were applied.

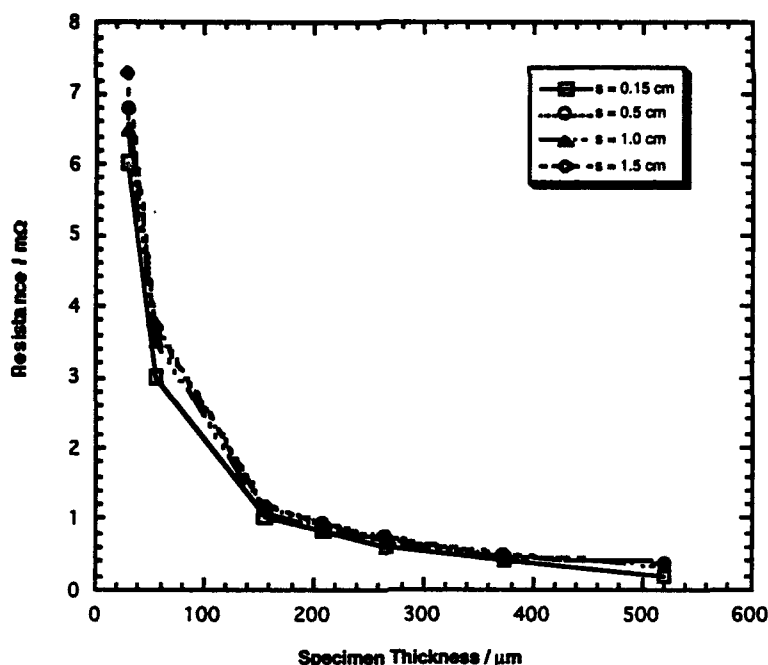


Figure 2. Dependence of the resistance of stainless steel (SS 316) plates (16x30 cm) on the thickness of the specimens, W. Four different probes (commercial Alessi and in-house fabricated probes) were used with contact spacing, s, of 0.15, 0.5, 1.0 and 1.5 cm, respectively. All probes satisfied the criterion: $W/s < 3$. Current pulsing technique for resistance measurements was used.

Table 2. Resistance values of SAE 4140 carbon steel discs (diameter = 9.7 cm) using constant DC and current pulsing techniques.

4 POINT PROBE	SPACING S/cm	SAMPLE THICKNESS W/cm	DC PULSING TECHNIQUE: RESISTANCE $\mu\Omega$	CONSTANT DC CURRENT: RESISTANCE $\mu\Omega$
In-house	1.0	1.385	4.44 \pm 0.15	3.3 \pm 0.2

Figure 3 shows four-point probe DC resistance measurements performed on SS 316 sheet (15 x 22 x 0.05 cm) at two different applied currents, 1 A and 10 A, respectively. Resistance values obtained at 1 A were systematically higher than those obtained if 10 A was passed through the current probes. This may indicate temperature compensation problems due to local overheating at high applied currents. No such differences in resistance were obtained on using the current pulsing technique when 5 A pulses with pulse duration of 15 and 25 ms, were applied. The majority of experiments were therefore performed using the current pulsing technique. The increase in resistance close to the edges of the SS 316 sample, as shown in Figure 3, reflected geometrical edge effects. These are discussed in more detail below.

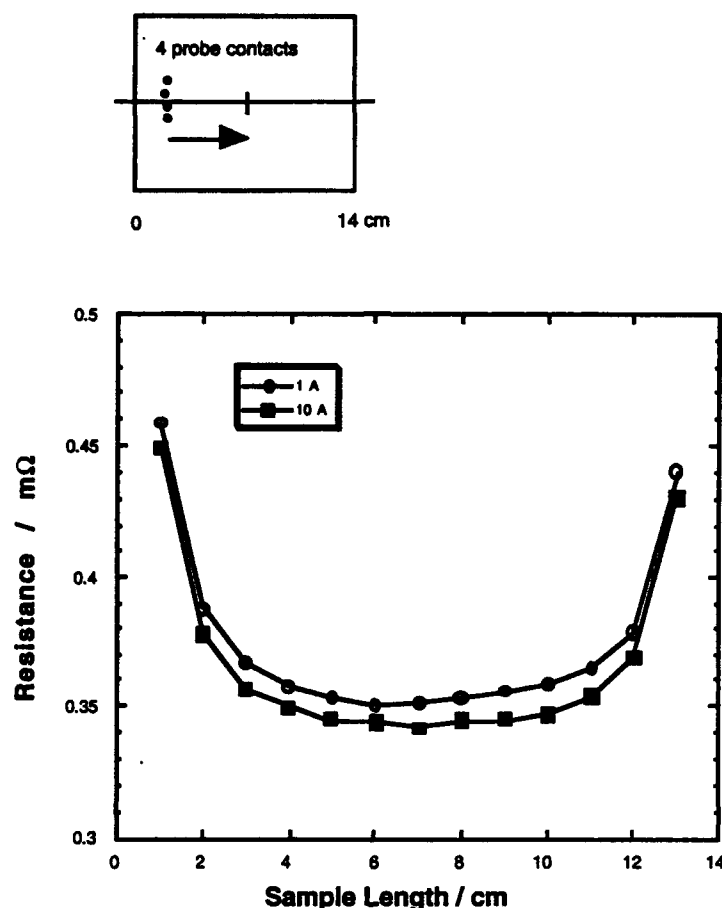


Figure 3. Constant DC current resistance measurements performed on a stainless steel (SS 316) sheet (15 x 22 x 0.05 cm) at two different applied currents. An in-house-fabricated four-point probe with a contact spacing of 1.0 cm was used. A sketch above the figure shows measuring positions on the specimen.

C. REPRODUCIBILITY OF RESISTANCE MEASUREMENTS BY THE PULSED CURRENT TECHNIQUE

Tables 3 and 4 summarize a statistical analysis of resistance measurements obtained by the pulsed current technique using Al and Alloy 625 plates. Reproducibility of the data: (a) with a single contact, i.e., with the probe pressed down on the sample and contacts kept continuously on the surface; and (b) with multiple contactings at the same position are compared. With increasing number of measurements, standard error and confidence intervals decreased. From Tables 3 and 4 it is possible to choose the required accuracy for resistance measurements by determining the number of measurements needed for each metal sample. The reproducibility of the measured electronic voltage signal where the probe was down, i.e., in continuous contact with the surface, was better than the reproducibility of electronic signals obtained where multiple contacting of the metal surface at approximately the same position was used. This indicates that contacting of a metal sample and, hence, design of a probe will be critical for developing an effective apparatus for nondestructive crevice corrosion detection.

Table 3. Statistical analysis of resistance measurements on Al plate (30.5 x 30.5 x 1.36 cm) derived from current pulsing technique.

	Resistance, Ohms														
Number of Times Probe Contacts Sample	1	5	1	10	1	20	1	30	1	40	1	50			
Number of Measurements	5	5	10	10	20	20	30	30	40	40	50	50			
Mean	6.56×10^{-7}	6.48×10^{-7}	6.90×10^{-7}	6.80×10^{-7}	6.60×10^{-7}	6.45×10^{-7}	6.43×10^{-7}	6.34×10^{-7}	6.37×10^{-7}	6.32×10^{-7}	6.33×10^{-7}	6.30×10^{-7}			
Minimum	6.30×10^{-7}	6.40×10^{-7}	6.40×10^{-7}	6.40×10^{-7}	6.30×10^{-7}	6.00×10^{-7}	6.10×10^{-7}	6.00×10^{-7}	6.10×10^{-7}	6.10×10^{-7}	6.00×10^{-7}	6.00×10^{-7}			
Maximum	6.70×10^{-7}	6.60×10^{-7}	6.90×10^{-7}	6.80×10^{-7}	6.90×10^{-7}	6.70×10^{-7}	6.70×10^{-7}	6.70×10^{-7}	6.60×10^{-7}	6.70×10^{-7}	6.80×10^{-7}	6.70×10^{-7}			
Standard Deviation	1.95×10^{-8}	8.37×10^{-9}	1.49×10^{-8}	1.25×10^{-8}	1.69×10^{-8}	1.96×10^{-8}	1.42×10^{-8}	1.87×10^{-8}	1.41×10^{-8}	1.76×10^{-8}	1.63×10^{-8}	1.73×10^{-8}			
Standard Error	8.71×10^{-9}	3.74×10^{-9}	4.72×10^{-9}	3.94×10^{-9}	3.77×10^{-9}	4.38×10^{-9}	2.60×10^{-9}	3.42×10^{-9}	2.23×10^{-9}	2.79×10^{-9}	2.31×10^{-9}	2.46×10^{-9}			
95% Confidence	1.71×10^{-8}	7.33×10^{-9}	9.26×10^{-9}	7.73×10^{-9}	7.39×10^{-9}	8.59×10^{-9}	5.09×10^{-9}	6.69×10^{-9}	4.38×10^{-9}	5.46×10^{-9}	4.53×10^{-9}	4.81×10^{-9}			
99% Confidence	2.25×10^{-8}	9.65×10^{-9}	1.02×10^{-8}	1.02×10^{-8}	9.72×10^{-9}	1.13×10^{-8}	6.70×10^{-9}	8.81×10^{-9}	5.76×10^{-9}	7.19×10^{-9}	5.96×10^{-9}	6.34×10^{-9}			

Table 4. Statistical analysis of resistance measurements on Inconel 625 alloy plate (15.2 x 15.2 x 0.657 cm) derived from current pulsing technique.

Resistance, Ohms													
Number of Times Probe Contacts Sample	1	5	1	10	1	20	1	30	1	40	1	50	50
Number of Measurements	5	5	10	10	20	20	30	30	40	40	50	50	50
Mean	45.74	45.68	45.7	45.68	45.61	45.64	45.61	45.57	45.49	45.55	45.58	45.52	45.52
Maximum	45.8	45.7	45.8	45.7	45.7	45.7	45.8	45.7	45.5	45.7	45.7	45.6	45.6
Minimum	45.7	45.6	45.6	45.6	45.6	45.4	45.6	45.3	45.4	45.4	45.4	45.3	45.3
Standard Deviation	5.48×10^{-2}	4.47×10^{-2}	4.71×10^{-2}	5.16×10^{-2}	3.08×10^{-2}	8.75×10^{-2}	4.34×10^{-2}	8.28×10^{-2}	3.62×10^{-2}	5.99×10^{-2}	4.95×10^{-2}	6.93×10^{-2}	6.93×10^{-2}
Standard Error	2.45×10^{-2}	2.0×10^{-2}	1.49×10^{-2}	1.63×10^{-2}	6.88×10^{-3}	1.96×10^{-2}	7.93×10^{-3}	1.51×10^{-2}	5.72×10^{-3}	9.47×10^{-3}	7.0×10^{-3}	9.90×10^{-3}	9.90×10^{-3}
95% Confidence	4.80×10^{-2}	3.92×10^{-2}	2.92×10^{-2}	3.20×10^{-2}	1.34×10^{-2}	3.84×10^{-2}	1.55×10^{-2}	2.96×10^{-2}	1.12×10^{-2}	1.86×10^{-2}	1.37×10^{-2}	1.94×10^{-2}	1.94×10^{-2}
99% Confidence	6.32×10^{-2}	5.16×10^{-2}	3.84×10^{-2}	4.21×10^{-2}	1.78×10^{-2}	5.05×10^{-2}	2.05×10^{-2}	3.90×10^{-2}	1.48×10^{-2}	2.44×10^{-2}	1.81×10^{-2}	2.56×10^{-2}	2.56×10^{-2}

D. DETECTION OF ARTIFICIAL CREVICES MACHINED IN ALLOY SAMPLES BY PULSED CURRENT RESISTANCE MEASUREMENTS

1. Recognition of Crevices by Measuring Changes In Sample Thickness by Resistance Method

Resistance of a metal sample will increase as the thickness of the sample decreases. Changes in specimen thickness due to artificially made crevices were measured by placing a four-point probe at the side opposite to where the crevice was made. A crevice (42.6 x 9.9 x 0.79 mm), ca. 12% of the specimen thickness, was machined in the center of an alloy 625 plate (15.2 x 15.2 x 0.657 cm) so that edge effects could be neglected. A probe spacing of 1.0 cm was used, satisfying the criterion, $W/s < 3$, for "sensing" the thickness of the specimen by resistance measurements. Figure 4 shows resistance data taken at 4 different positions surrounding the crevice and one (#5) taken directly over the crevice. The four-point probe was positioned perpendicularly to the edge of the crevice (cf., schematic adjacent to Fig. 4). Twenty resistance values per contact point were taken and measurements were repeated 3 times. Resistance values were reproducible within $\pm 0.1 \mu\Omega$.

Resistance values measured at positions #1 and #2 (ca. 1 cm away from the crevice) were similar, because they were not influenced by the crevice.

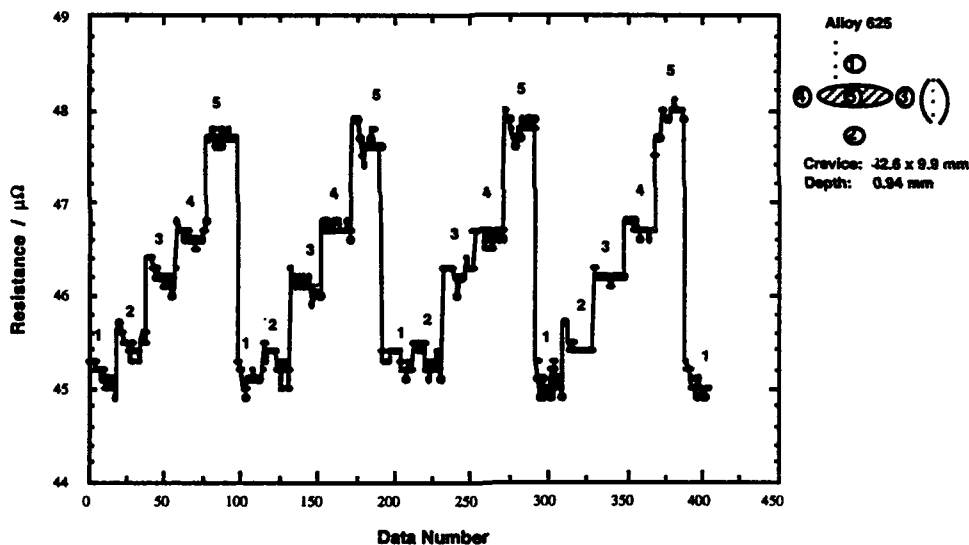


Figure 4. Changes in resistance around a crevice machined in an Inconel Alloy 625 plate measured by pulsed current four-point probe resistance method.

At these positions, current lines passing through the specimen, applied by the two outer probes, do not pass near, or through, the crevice, and, thus, the change in thickness was not sensed by the voltage probes. On the contrary, at positions #3 and #4 (ca. 2 cm away from the crevice), the applied electric field penetrates the crevice and an increase in resistance was observed. At position #5, directly above the crevice, a significant increase in resistance, ca. $1.5 \mu\Omega$, was measured. This experiment clearly demonstrates the ability to detect a crevice by measuring an increase in resistance. If the probe was positioned in parallel to the edge of the crevice (cf., Fig 5) the same resistance pattern was observed at positions #1 through #5 as when the probe was placed perpendicularly to the edge of the crevice (cf., Fig. 4).

These experiments illustrate that the existence of a crevice can be detected by the four-point probe resistance method, using a pulsed current, even if the resistance is not probed directly over the position of the crevice, but in the vicinity of the crevice.

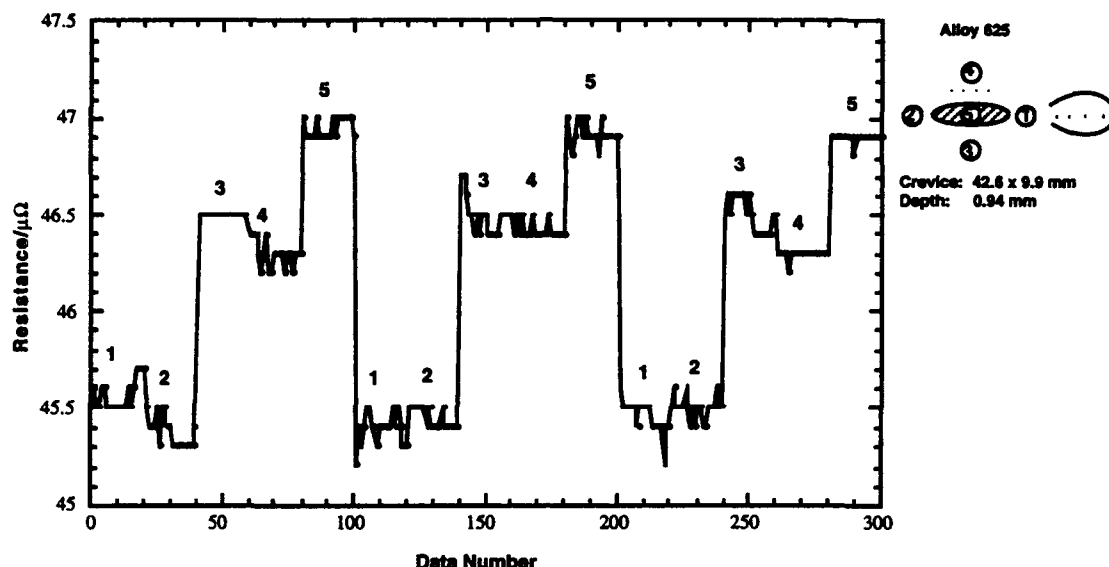


Figure 5. Same as Figure 4 except that the probe was positioned in parallel to the crevice.

2. Dependence of Resistance on the Depth of a Crevice

Crevice, 45 x 6 mm, having increasing depths, and placed 18 mm apart, were machined in an Al plate (30.5 x 30.5 x 1.36 cm). Resistance values were measured at positions surrounding crevices and directly above the location of crevices. The probe was positioned in parallel to the crevices. A sketch of the positions where the measurements were taken is shown in the schematic at the top of Figure 6. Figure 6 shows increases in resistance that were observed with increasing crevice depth. In Figure 7 this relationship is presented more quantitatively. A nonlinear relationship between resistance and crevice depth was obtained because the ratio between the specimen thickness and the probe spacing was greater than 0.5 (0.68), thus corresponding to Equation (4), where a departure from linearity is determined by the geometrical correction factor, CF. At positions removed from the locations of crevices (cf., #1, #4, #7, #10, #13, #16, #19, #22, #25 and #28, in Fig. 6) resistance values were constant and reflected the total thickness of the specimen. It is noteworthy that, at locations between the crevices (cf., #3, #6, #9, #12, #15, #18, #21, and #24, in Fig. 6) resistance values also increased, reflecting the fact that the measurements were taken in the vicinity of crevices. Compared to these locations, the resistance value at position #27 was lower because that location was surrounded by only one crevice (#26).

Experiments described in this section demonstrate that due to precise directioning of a localized electric field through a metal specimen, an in-depth profile of crevices and their spatial resolution can be achieved by means of four-point probe resistance measurements.

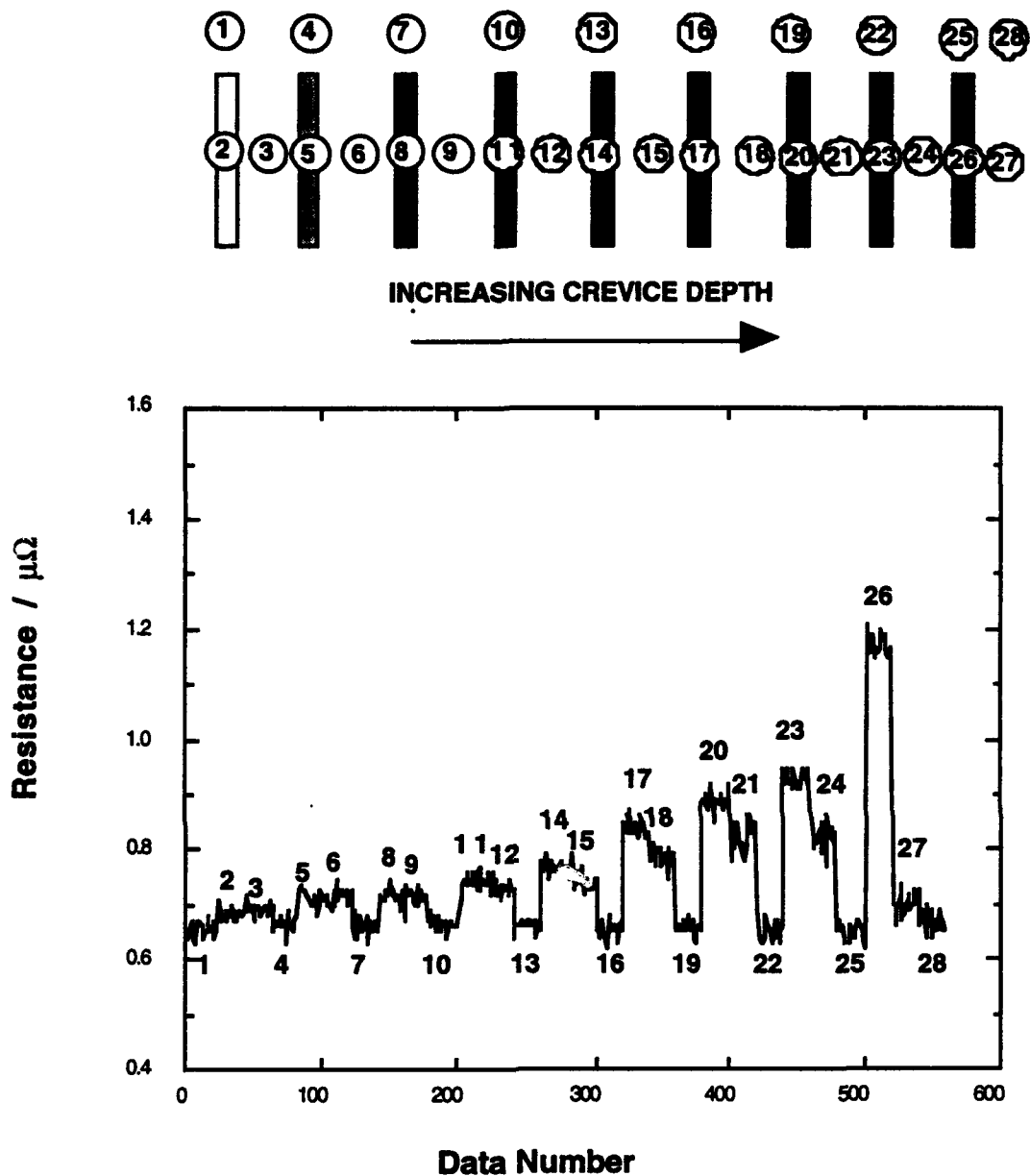


Figure 6. Detection of crevices of various depths by pulsed current resistance measurements. Locations of crevices and other locations where resistance measurements were made are indicated in the sketch at the top of the Figure. Twenty resistance values per location were measured. An in-house fabricated probe with a spacing of 1.0 cm between the contacts was used.

3. Nondestructive Detection and Imaging of Crevice Patterns

Once a number of statistically significant resistance values were obtained per contact location for a specific material, an image of a crevice could be created by computerized data acquisition/processing of resistance values sampled at different locations

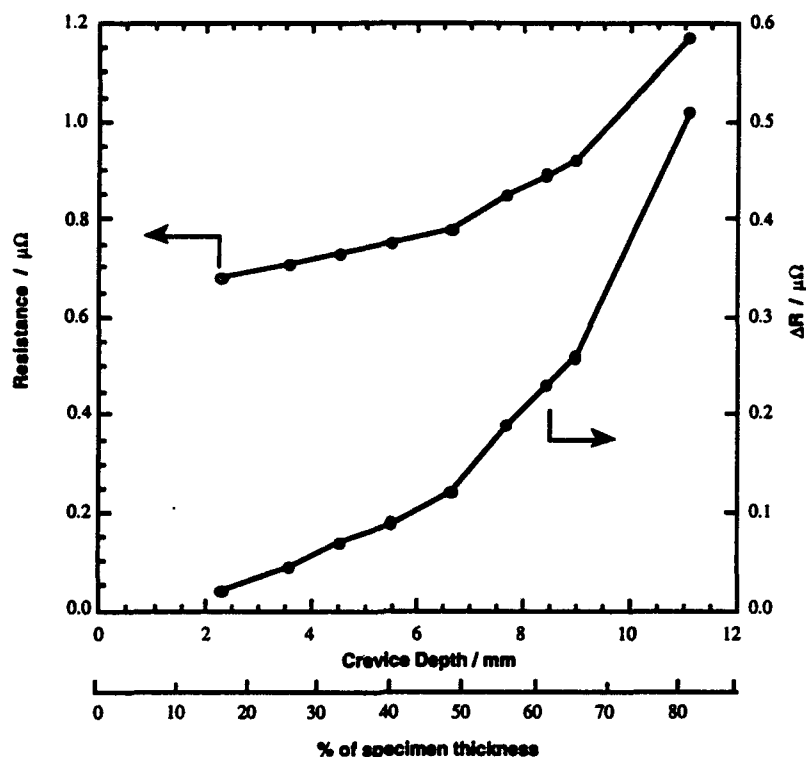


Figure 7. Dependence of resistance on crevice depth as obtained from the data presented in Figure 6. Crevice depth is indicated both in mm and as a percentage of specimen thickness. Differential resistance, ΔR , was obtained by subtracting a "background" resistance, measured at locations not affected by the presence of crevices.

along the specimen surface. Figure 8 shows an image of a crevice (42.6 x 9.9 x 0.79 mm) obtained from pulsed current resistance values for an Alloy 625 plate (15.2 x 15.2 x 0.657 cm). Resistance values were measured on the surface of the plate opposite to that of the crevice with a spatial resolution of 1.0 cm. The image was obtained by projecting a 3-dimensional picture of resistance values on the x-y plane. Although a low spatial resolution between contact locations was used, Figure 8 reveals a clear image of the crevice located in the center of the Alloy 625 sample. An increase in resistance close to the edges of the plate is due to the use of data uncorrected for geometrical factors. It must be noted that the lines of equal resistance close to the edges will be severely disturbed by the presence of an eventual crevice that could be easily detected.

Figure 9 is a 3-dimensional image of a crevice (43 x 16.3 x 4.35 mm, and having a depth corresponding to 32% of total specimen thickness) machined in an Al plate (30.5 x 30.5 x 1.36 cm). Spatial resolution of the measurements was 0.5 cm. Comparing values of resistance measured directly above the crevice, ca. 0.72 - 0.77 $\mu\Omega$, with resistance values for crevices of various depths (cf., Figs. 6 and 7), the depth of the crevice, ca. 4 - 5 mm was obtained. This value is in good agreement with the exact crevice depth of 4.35 mm.

Using computerized data acquisition of four-point probe resistance values measured along a metal specimen, with a desired spatial resolution, an accurate 3D-image of a crevice pattern can be obtained for both aluminum and alloy 625.

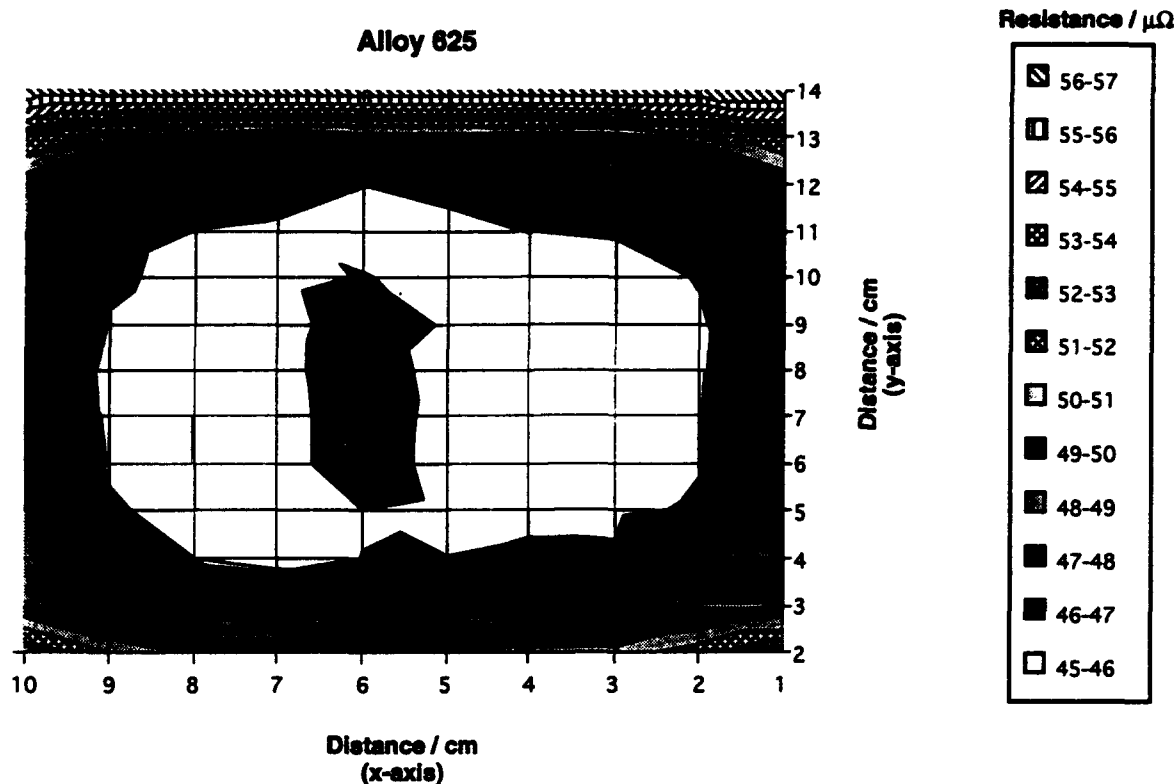


Figure 8. Image of a crevice (dark shaded area in the middle of the specimen) obtained from pulsed current resistance measurements using computerized data acquisition/processing.

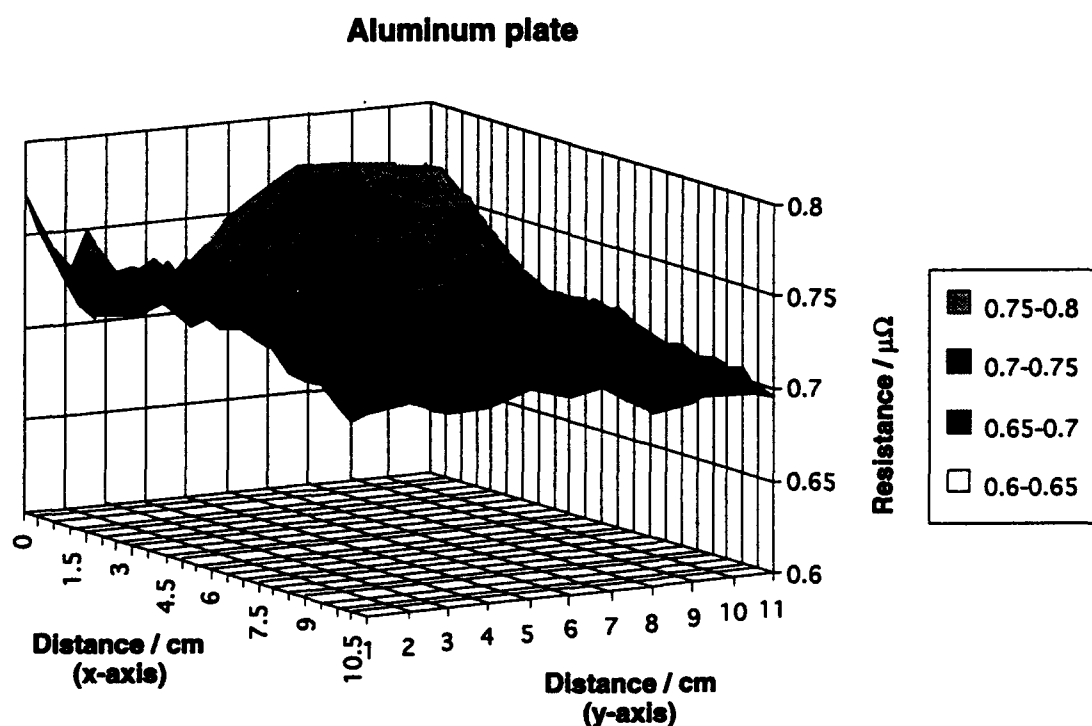


Figure 9. A 3-dimensional computerized image of a crevice obtained by pulsed current resistance measurements. The crevice is seen as an increase in resistance (light-gray areas) above the "background" resistance (obtained at regions having no crevice). 10 resistance values per contact location were taken.

4. Crevice Size and Depth Detection Limits of Pulsed Current Resistance Measurements

An attempt was made to determine the minimum size and depth of crevices that can be detected by the present experimental setup. It was shown in the previous Section that relatively large crevices, 42.6 x 9.9 mm in alloy 625 and 45 x 7.36 mm in aluminum, but low in depth, ca. 12% of total specimen thickness for alloy 625 (cf., Fig. 9) and 22% for aluminum plate (cf., Fig. 7, position #2, and Fig. 8), could be easily detected with the experimental setup described in Section II. Figure 10 shows resistance data obtained at locations surrounding a crevice and directly above a crevice machined in aluminum, approximately 7 times smaller than that described in Fig. 6, position #2, and having a depth corresponding to 44% of the specimen thickness. A commercial probe (Ultraoptec, Inc.) with a spacing between contacts of 9 mm was used. Although the data obtained is more noisy, because the crevice was smaller than the contact spacing, an increase in resistance can be seen at the location of the crevice. The same experiment was repeated with a commercial probe of smaller spacing, $s = 1.5$ mm, which did not sense the thickness of the crevice (cf., Fig. 11.). This experiment clearly demonstrates the need to adjust the probe contact spacing, so as to satisfy the criterion $W/s < 3$, and/or apply a higher current range in order to increase voltage probe sensitivity and accuracy (see Section IV).

Because of higher reproducibility and the greater ability of contacting alloy 625 surfaces (cf., Table 4), crevices of smaller size were detectable in alloy 625 than in aluminum. Figures 12 and 13 demonstrate the recognition of crevices made in a 625 alloy plate having dimensions of 6.4 x 9.9 x 1.54 mm (i.e., having a depth corresponding to 23 % of the specimen thickness) and 2 mm dia x 3.15 mm (i.e., having a depth corresponding to 46% of the specimen thickness). *For the crevice of only 2 mm in diameter and having a depth corresponding to 46% of the specimen thickness, a resistance change of ca. $0.3 \mu\Omega$ was clearly detectable on alloy 625 with the present experimental setup.*

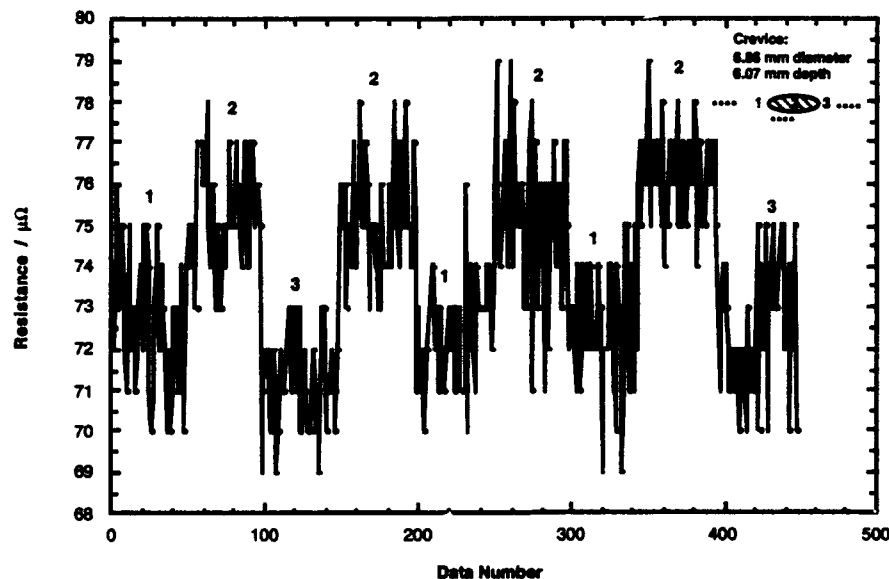


Figure 10. Detection of a small crevice by monitoring an increase in resistance at the crevice location. A crevice, machined in an Al plate (30.5 x 30.5 x 1.36 cm), was located at position #2 (see insert). Twenty resistance values per contact location were measured. A commercial probe (Ultraoptec, Inc.) with a spacing between probe contacts of 9 mm was used.

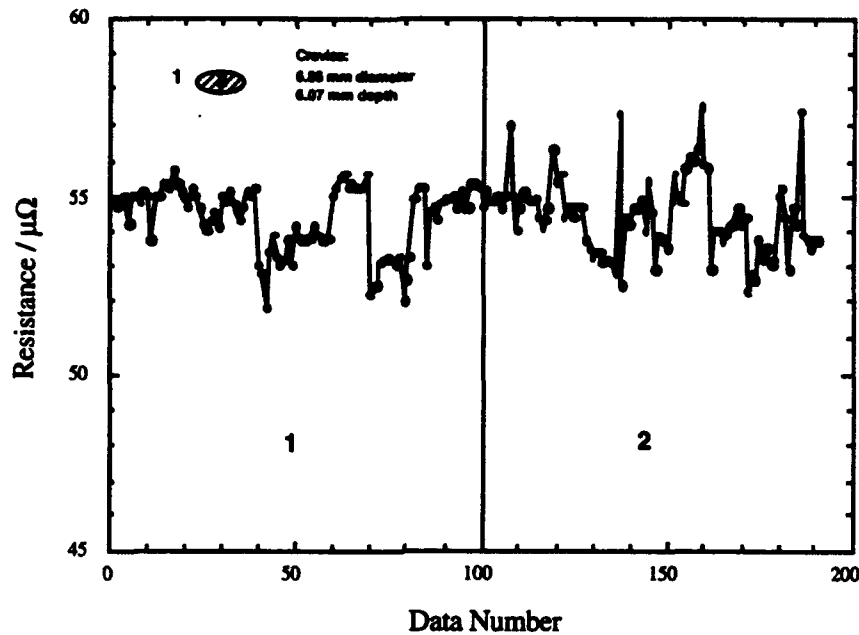


Figure 11. Same as in Figure 10 except that the probe (Ultraoptec, Inc.) had a contact spacing of 1.5 mm. Ten resistance values were taken with the probe contacts pressed on the specimen surface; the probe was then lifted and the measurements were repeated. This sequence was repeated 10 times (total number of resistance values = 100) for locations #1 and #2 (shown in the insert of the Figure). No measurable increase in resistance was observed at the crevice (location #2) because the criterion: thickness/spacing < 3 was not satisfied.

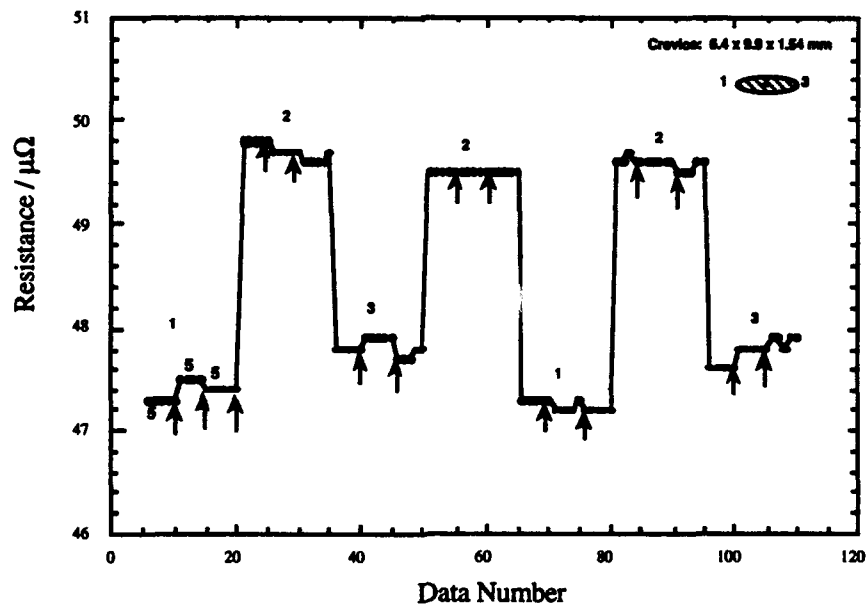


Figure 12. Detection of a small crevice (located at position #2, see insert), having a depth corresponding to 23 % of an Inconel alloy 625 (15.2 x 15.2 x 0.657 cm) specimen thickness. Fifteen resistance values per contact location were measured with the probe raised each time after a set of 5 readings was taken (see arrows). An in-house fabricated probe with a spacing between probe contacts of 10 mm was used.

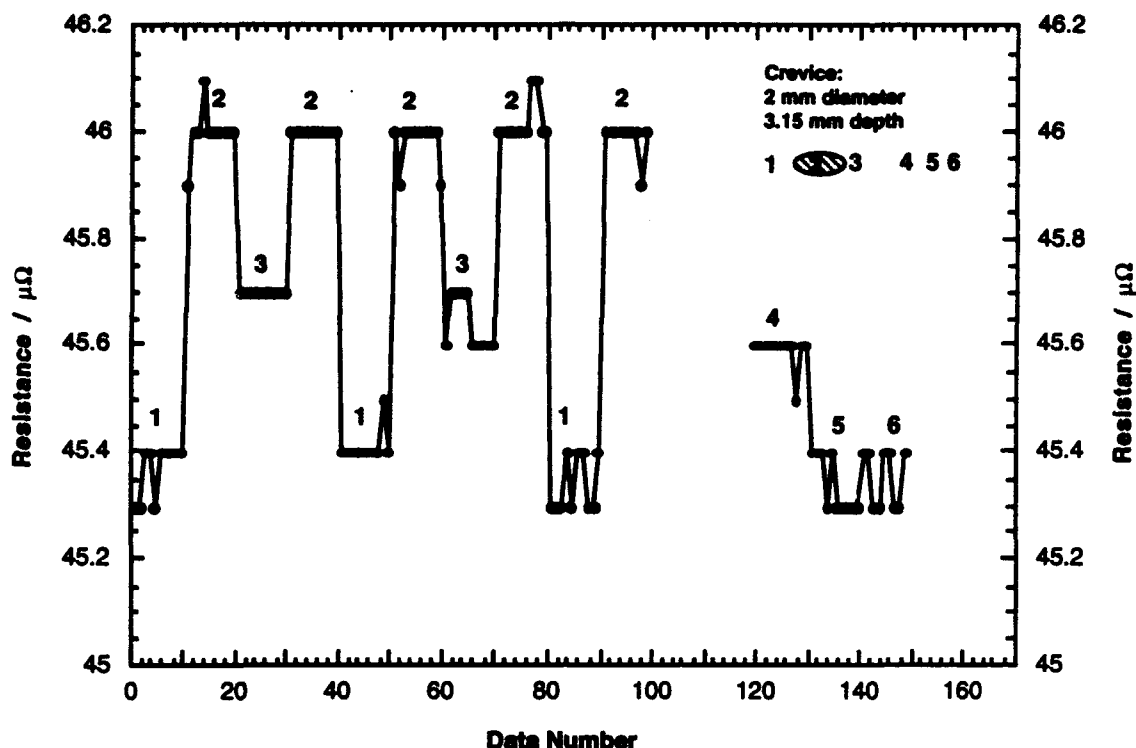


Figure 13. Changes in resistance nearby and directly above the smallest crevice detected (position #2, see insert) for an Inconel alloy 625 specimen (15.2 x 15.2 x 0.657 cm). Ten resistance values per contact location were measured with an in-house fabricated probe having a spacing between the probe contacts of 10 mm. At locations 4, 5 and 6, a "background" resistance, far from the crevice, was measured. An increase in resistance of ca. 0.2 $\mu\Omega$ was observed directly above the location of the crevice (#2).

5. Detection of Crevices in Ring-Shaped Specimens

It was mentioned earlier that if the resistance was measured along lines parallel and close to the edge of a metal specimen, the presence of a crevice close to the edge of the specimen (cf., Fig. 9) could be detected without correcting the resistance data for edge effects. Figure 14 shows a 3-dimensional image of a crevice (11.4 x 6.3 x 3.56 mm, having a depth corresponding to 27% of the thickness of the metal ring), machined in a SS 316 ring (of inner diameter = 10.8 cm, outer diameter = 17.8 cm and 12.88 mm thick), obtained by the pulsed current resistance method. Measurements were made along a series of circular paths, ca. 2.5 mm apart and with spatial resolution along each circular path of 8 mm. A special holder for the four-point probe was designed to facilitate movement of the probe along circular paths, i.e. at constant radii. Resistance values were the same, within the accuracy of the measurements, along each circular path. At the location of the crevice a significant increase in resistance was observed where each circular path intersected the crevice. Along circular paths located further away from the center of the ring, where the crevice did not extend, no change in resistance was observed (cf., Fig. 14). *Thus, for any metal having a crevice located at the edge of the specimen, measurement of the electrical resistance along well defined geometrical pathways can reveal the presence of a crevice by an increase in resistance beyond that measured as a "background" value.*

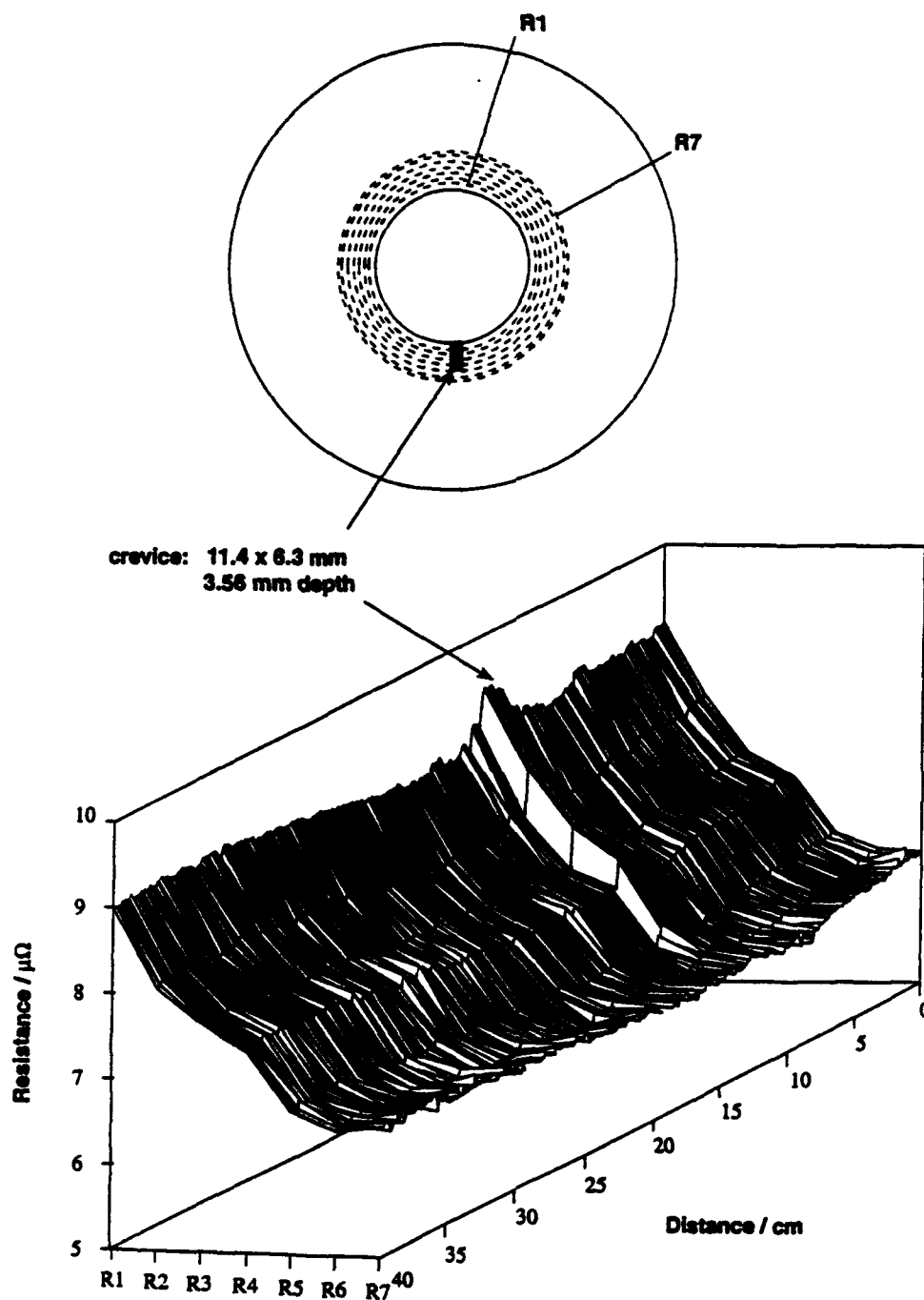


Figure 14. Three-dimensional image of a crevice, machined in a SS 316 ring obtained by pulsed current resistance measurements. Resistance values were measured along circular pathways (see drawing above the three-dimensional image), ca. 2.5 mm apart and with spatial resolution along each circular path of 8 mm.

E. DETECTION OF ARTIFICIAL CREVICES ON CARBON STEEL FLANGES

1. Recognition of Crevices by Resistance Measurements Along Circular Pathways on Flanges

The geometrical shape of real flanges significantly affect nondestructive methods for the detection of crevice corrosion. Resistance measurements have the advantage that the correction factor for edge effects can be theoretically predicted and calculated for different geometrical shapes, including that of a flange complicated by screw holes and different thicknesses at different parts of the flange. In Section D.5. it was demonstrated that the presence of a crevice can be detected on metal rings without correcting resistance data for geometrical factors, i.e., for edge effects. In this Section, similar results will be presented for a class 150 carbon steel pipe flange with a weld neck (Weldbend), designed for pressures of 150 psi and accepting 1.5" diameter tubing. In Figure 15 a drawing of the flange is presented together with the locations of crevices. Also, locations where resistance measurements were made are marked. A special four-point resistivity probe holder (cf., Fig. 16) was fabricated facilitating movement of the probe along circular paths on the surface of the flange. In all the experiments performed with the flange, the four contacts of the resistivity probe were positioned not in a straight line, but in an arc corresponding to a circular pathway along which measurements were made. Thus, resistance measurements could be performed on circular pathways of various radii (cf., Fig. 15, dark shaded ring), which is critical for the detection of crevice corrosion.

Crevice were made at locations where crevice corrosion is expected to occur in a real situation, i.e., at the rim where the gasket seals the flange. Figure 17 shows resistance data obtained at crevice #1 (6.70 mm diameter, 10.72 mm deep, ca. 48% of flange thickness, cf., Fig. 16) drilled at position 2, and at three other geometrically equivalent positions: 6, 10 and 14 (cf., Fig. 15). All four positions were considered geometrically equivalent because they were located at the same radial distance (see dark shaded ring, Fig. 15) and their distances from the screw holes were the same. Compared to the approximately uniform resistance values measured at positions 6, 10 and 14, which were far removed from a crevice, an increase in resistance of ca. $0.35 \mu\Omega$ directly over the crevice (position 2) was clearly observed. As shown in Figure 17, resistance measurements were repeated three times (with 20 values collected per contact position), and excellent reproducibility was obtained. Small "resistance peaks", observed each time the probe position was changed (cf., Fig. 17), was investigated more thoroughly and is discussed in Section E2.

Figure 18 shows the detection of the same crevice #1 with resistivity probes having the same distance between the current contacts, $s_i = 60$ mm, and varying distance between the voltage probes. The crevice was clearly detectable with the probe having equal spacing between all four contacts and for the probe having a large spacing between the voltage contacts ($s_v = 25$ mm). The resistivity probe with the smallest spacing between the voltage contacts, $s_v = 7$ mm, yielded the least resolution in resistance, probably because the ratio W/s_v was slightly higher than 3 (3.19). As expected, absolute resistance values were different for all three cases.

Figure 19 shows changes in resistance at positions around and directly over the smallest crevice detected (cf., Fig. 15, crevice #2 at position 15) in the flange by the present experimental setup.

Dimensions of the smallest crevice detected in the flange analyzed were: 2 mm in diameter and a depth corresponding to 44% of the flange thickness.

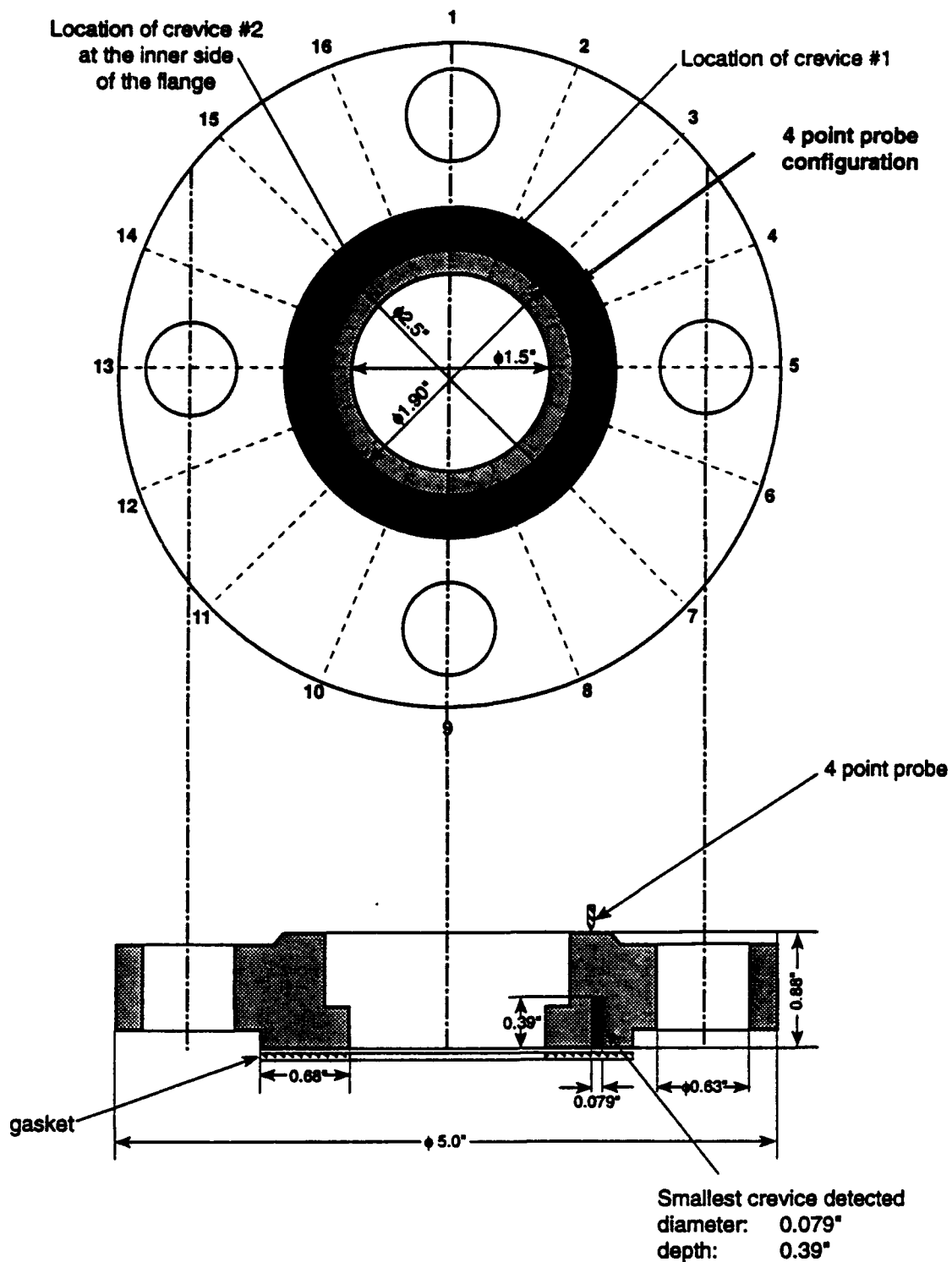


Figure 15. Schematic diagram of flange used in tests of crevice detection by resistance measurements. Crevice locations (#1 and #2) as well as locations where "background" resistance measurements were made are indicated by lines 1 to 16.

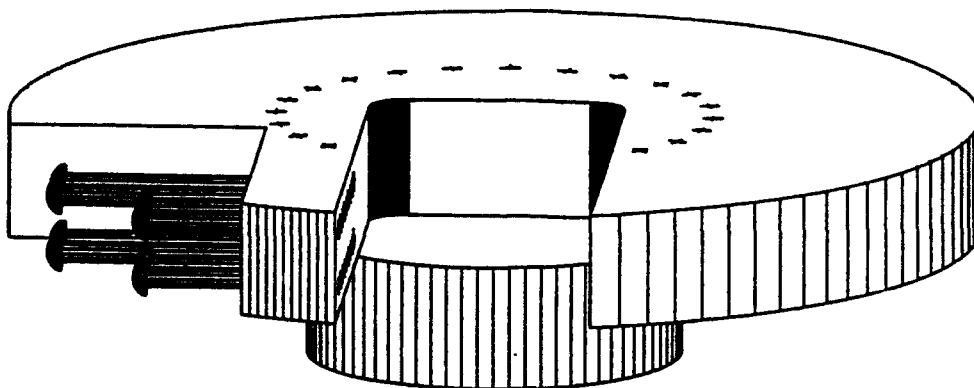


Figure 16. Four-point probe holder for resistance measurements on a flange along circular paths of constant radii.

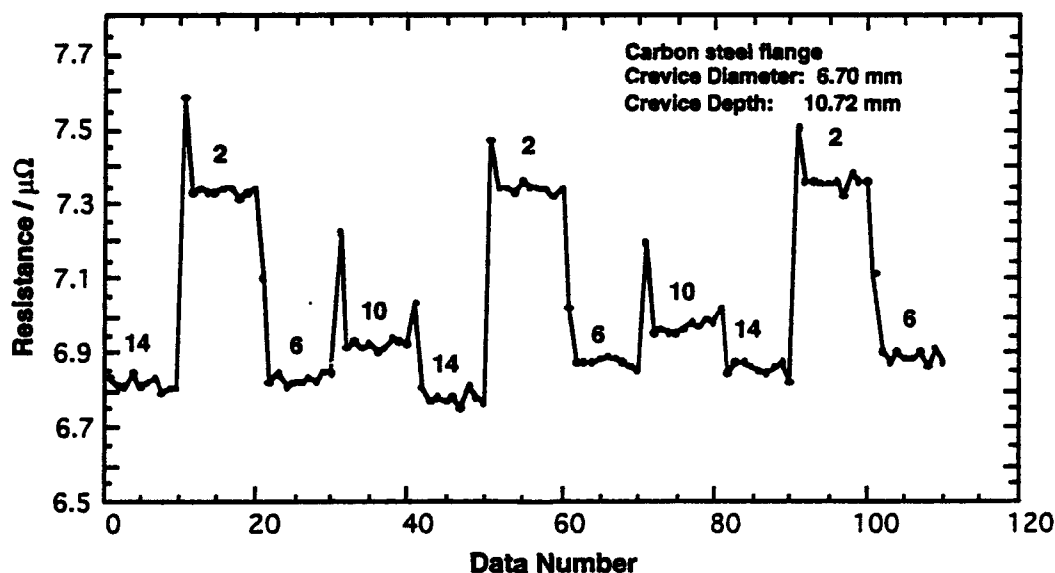


Figure 17. Resistance values obtained at crevice #1 artificially made in a flange shown schematically in Fig. 15 (position #2, 6.70 mm in diameter, 10.72 mm deep, ca. 48% of the flange thickness), and at three other geometrically equivalent positions: 6, 10 and 14 (cf., Fig. 15).

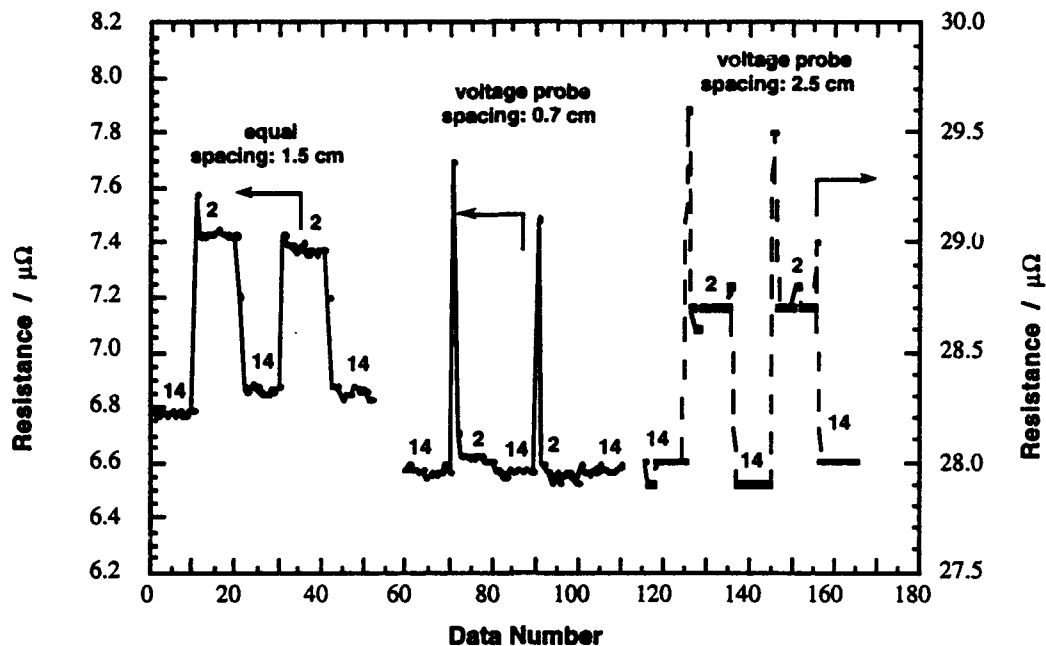


Figure 18. Detection of crevice #1 drilled in a flange (cf., Figs. 15 and 17), using four-point probes having the same distance between current probes, $s_i = 55$ mm, and varying distance between the voltage probes. Numbers on the profiles indicate locations where resistance measurements were made.

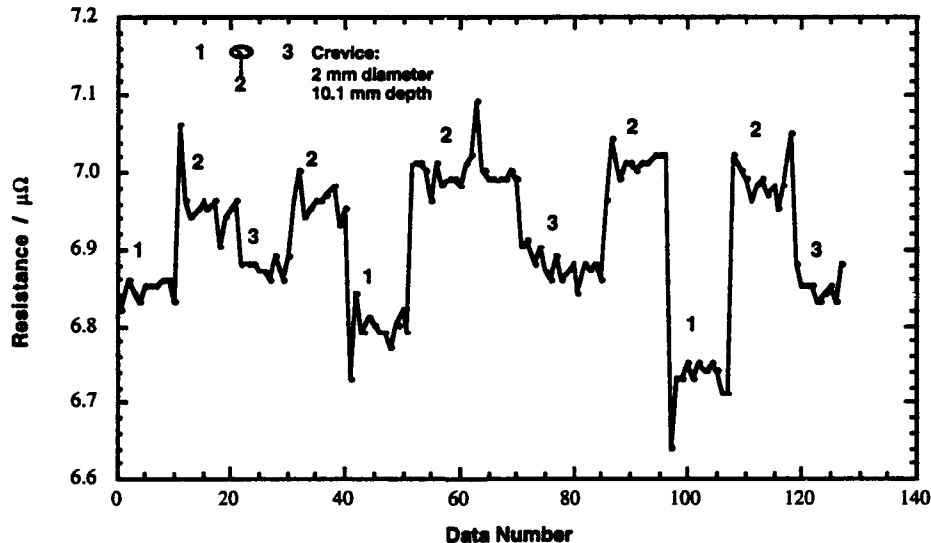


Figure 19. Resistance data for the smallest crevice detected (cf., Fig 15, crevice #2, measurement position 15) on a class 150 carbon steel flange. The crevice was only 2 mm in diameter and had a depth corresponding to 44% of the flange thickness. Numbers on the profile indicate locations where resistance measurements were made.

2. Observation of Resistance "Peaks" by Application of Current Pulses Through Carbon Steel Flange

Resistance data shown in Figures 17 and 18, for a carbon steel flange, exhibited small "peaks" each time the probe was moved from one position to another distant position. These "peaks" were observed only after the first current pulse was applied at each new location. Their origin was investigated in terms of the presence of impurities at the surface. A thorough cleaning and preparation of the flange surface did not affect their occurrence. These "peaks" were observed only on carbon steel samples and not on stainless steel (SS 316) or alloy 625. This led to the conclusion that the sudden increase in resistance could be a consequence of an induced alignment of magnetic dipoles during the first current pulse applied between current contacts at each new probe location. According to Ampere's law (which defines a dependence between the current and an induced magnetic field), this could be a feasible explanation. In order to prove this hypothesis an experiment was performed on the same flange which involved reversing the current probe polarity and measuring resistance in the presence and absence of a permanent magnet. Results are shown in Figure 20. Ten resistance values per probe position were measured.

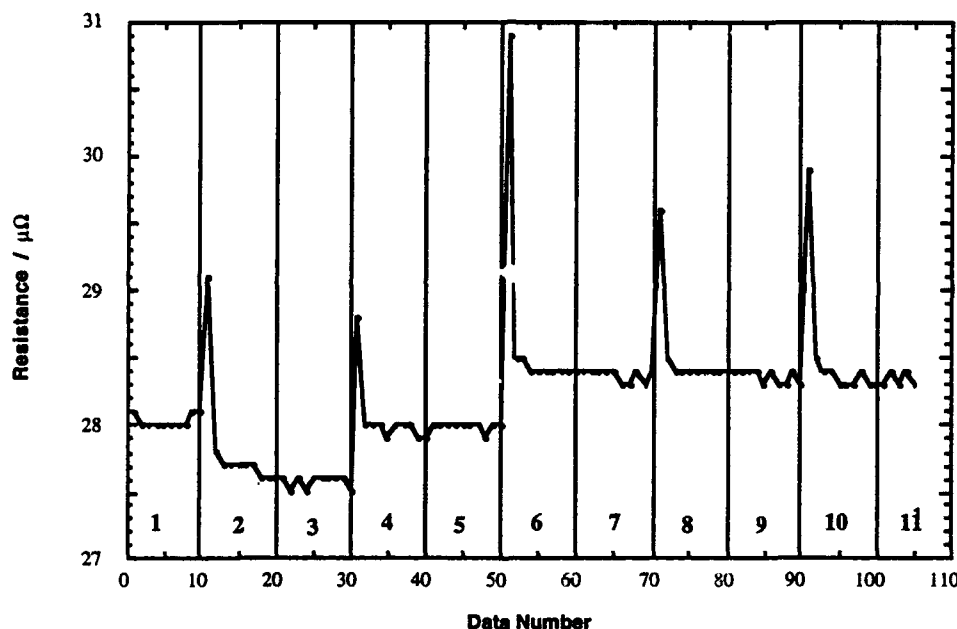


Figure 20. Investigation of the origin of resistance "peaks" observed after the first current pulse was applied at a new probe location. Different conditions were tested: (i) polarity of probe current switched (at #6); (ii) a permanent magnet placed in the vicinity of the measurement location (#8); and (iii) switching the magnet polarity (#10). Measurements were performed on the flange at geometrically equivalent positions 10 and 14 (cf., Fig. 15).

Measurements were made at two geometrically equivalent positions on the flange (10 and 14) where no crevices existed. A resistance "peak" occurred only after the first current pulse, between data sets #1 (taken at position 10) and #2 (taken at position 14), presumably because of rapid alignment of local magnetic dipoles caused by a current pulse. If the probe was lifted up and pressed again at the same position (see data between #2 and #3) no "peak" was found in the first current pulse applied, indicating that local magnetic dipoles were already aligned according to the direction (and amplitude) of the very first current pulse at that location. This sequence was repeated for data sets #4 and #5, taken at position 10. Between data sets #3 and #4 a resistance "peak" occurred again because of

alignment of dipoles at the new location, and was absent between #4 and #5 when the probe was only lifted up and pressed back down at the same location.

While maintaining the same position (10), the current polarity was then switched between recording data sets #5 and #6. A large peak appeared due to the change in current direction and new polarization conditions. The time for alignment of local dipoles was obviously very short, lasting only during the first approximately 25 ms of a current pulse and not affecting the rest of the data taken at the same position. Once alignment of dipoles at a new location was accomplished subsequent current pulses did not induce further alignment at that location (cf., between data sets #2 and #3, #4 and #5, #6 and #7, #8 and #9 and #10 and #11).

Between data sets #7 and #8, a permanent magnet was placed close to the position where measurements were taken, and this resulted in a similar resistance "peak" (cf., Fig. 20). If the poles of a magnet were switched the resistance "peak" was observed again. This sequence of resistance data, demonstrates that a rapid alignment of local magnetic dipoles can occur when using pulsed current resistance measurements on ferromagnetic materials. *However, in the detection of crevice corrosion, the problem of alignment of local magnetic dipoles by large amplitude current pulses can be simply avoided by neglecting the first data point at each new location measured. This effect was not observed on nonferromagnetic materials, such as alloy 625, used by the Navy as a material for gasketed flanges for sea-water pipelines.*

The results shown in Figure 20 initiated a brief investigation of the possible use of monitoring changes in the magnetic field of a coil placed in the vicinity of a flange for the non-destructive detection of crevices in a flange. This method is based on changes in the impedance of a coil, specifically its inductance, due to perturbation of a magnetic field penetrating the flange at crevice locations. Changes in the inductance of a small coil were therefore monitored by scanning the surface of the flange shown in Fig. 15 by an inductance meter. It was found that the inductance dramatically changed when the coil was placed in the vicinity of the flange, showing the presence of a ferromagnetic material, but the changes were too small to detect crevices in the flange. Also, large diameter screw-holes could not be detected, showing a shallow depth of penetration of the magnetic field and its low selectivity compared to the application of an electric field in resistance measurements. This method, if shown to be successful, could be used only with ferromagnetic materials, and not with stainless steels or Inconel 625, which have a low percentage of iron or other paramagnetic elements.

IV. CONCLUSIONS AND RECOMMENDATIONS

During the Phase I project, Lynntech, Inc, demonstrated the feasibility of resistance measurements as the basis of a non-destructive method for the detection of crevice corrosion in gasketed flange materials. *Using laboratory scale test systems, the feasibility of using a pulsed current resistance measurement method for the detection of artificially made crevices in geometrically simple metal specimens, e.g., plates and rings, as well as in real flanges, was demonstrated.*

Resistance measurements obtained by application of constant DC currents and pulsed current methods were compared. It was found that the pulsed current method exhibited the following advantages compared to the constant DC method:

- a) No local overheating, thus changes in resistance with time were not observed as compared to constant DC current measurements.

- b) Large currents could be passed through metal samples with no significant temperature changes of the samples, thus higher sensitivity for resistance measurements was obtained.
- c) Thermal energy introduced into a sample by a current pulse can be controlled by regulating the pulse amplitude, pulse duration and time between pulses. Thus, the pulsed current resistance measurement method could be used in a repetitive way giving rise to enhanced reproducibility and accuracy of the results compared to the constant DC method.

The results obtained using the *pulsed current resistance measurement method* for the detection of crevices of different sizes, artificially made in various materials, are summarized below:

1. Reproducibility of voltage signals measured on applying repetitive pulsed currents with the four-point probe in continuous contact with the surface of a metal, was better than the results obtained when repetitive contacting of metal samples was employed. This indicates the importance of the probe design for the non-destructive detection of crevice corrosion by the pulsed current resistance measurement technique. More accurate measurements were obtained on Alloy 625 compared to aluminum, because of the lower reproducibility of contacting aluminum due to the presence of an oxide film on the surface.
2. By adjusting the spacing between the probe tips, i.e., satisfying the ratio: specimen thickness/spacing < 3, changes in specimen thickness due to the presence of a crevice could be clearly detected by an increase in resistance at the crevice. Measurements were made on specimens on the side opposite to that where crevices were machined.
3. The existence of an artificially made crevice could be detected even if the resistance was not probed directly above the crevice, but in the vicinity of the crevice. This depended on the direction and penetration of the electric field applied through the current probes.
4. Since an electric field can be applied locally in a precise manner, by passing current pulses through a probe, an in-depth profile and spatial resolution between crevices could be achieved by this method.
5. Probing the resistance of a metal sample along defined pathways for samples having various geometric shapes, can reveal the presence of a crevice, even without correcting the resistance data for edge effects. This made possible the detection of crevices on ring-shaped samples and on real flanges.
6. Using computerized data acquisition and processing, three-dimensional images of crevices, artificially made in aluminum, carbon steel, stainless steel (SS 316), and Inconel Alloy 625 specimens of various geometries, were obtained with accurate spatial (ca. 2 mm) and depth (ca. 11% of specimen thickness) resolution.
7. Crevices of different sizes and depths were detected on class 150 carbon steel flanges. The smallest crevice detected, with the present experimental setup, were only 2 mm in diameter and had a depth corresponding to 44% of the flange thickness.

Theoretical internal bursting pressures for tubular products can be calculated from Barlow's formula:

$$p = 2 St/D$$

(6)

where p = bursting pressure; S = yield strength, ca. 75,000 psi for stainless steels; t = wall thickness and D = outside diameter. Although a minimum flange thickness needed to avoid flange destruction can be theoretically calculated, e.g., from Equation (6), a safe working pressure is generally taken as 25% of the theoretical bursting pressure¹³. Correspondingly, a critical reduction in flange thickness caused by corrosion, which can be tolerated, is also ca. 25 %. This value concerns the change in thickness along the whole diameter of the flange. The area occupied by crevices made in a flange investigated in this project covered only 0.14 - 1.3% of the total surface area of the flange subject to corrosion. The capability to detect damage of that size (ca. 0.1 - 1%) and depth (10-40%) seems very promising for the development of an apparatus which will be able to non-destructively detect critical crevice corrosion damage on a flange which needs to be replaced.

8. Changes in impedance (inductance) of a coil, placed in the vicinity of a flange, were investigated in order to detect crevices by perturbation of the magnetic field of the coil penetrating the flange. Preliminary data showed a shallow depth of penetration of the magnetic field and a low selectivity for non-destructive crevice corrosion detection compared to the application of an electric field associated with resistance measurements. This magnetic field-based method, if shown to be successful, could be used only on ferromagnetic materials, while resistance measurements could be used on any flange material, including Inconel alloy 625.

On the basis of results obtained in the Phase I project, advantages of using the pulsed current resistance technique for the non-destructive detection of crevice corrosion are outlined in Table 5.

Table 5. Advantages of four-point probe pulsed current resistance measurements for nondestructive detection of crevice corrosion in gasketed flange materials.

- very simple and straightforward method: resistance measurement theory is well developed and understood
- resistance measurements based on current pulsing are more sensitive than measurements made using constant DC currents because they are less subject to overheating at high applied currents
- electric field penetration through a metal specimen, determined by the position of the current probes, allows for easy determination of crevice depth profiles and their sizes
- easily automated and computerized technique, which can provide high resolution images of flange damage
- low development and operational costs
- because of their small dimensions, probes can be easily adapted and maneuvered on the outer side of gasketed joints for their inspection

The work outlined in this report is based on the application of high amplitude pulsed currents for accurate resistance measurements on gasketed pipeline joints. In order to apply this technique to real pipeline systems, for the rapid and efficient detection of crevice corrosion in flanges, several *recommendations and possible future directions* for improving the technique in a Phase II follow-on project are outlined below:

1. Results obtained in the Phase I work fully demonstrate the feasibility of using the pulsed current resistance technique for the non-destructive detection of crevice corrosion in flange materials. Thus, the main objective of a follow-on Phase II project should involve full development of an automated, portable, **flange crevice corrosion detection system**, consisting of: (i) resistance probes of appropriate size; and (ii) a computerized resistance meter based on high amplitude pulsed currents/low voltage sensing system. The work in Phase II should concentrate on designing and fabricating the detection system and evaluating its performance exclusively on real, corroded flanges.
2. Improvements in the four-point probe design should include:
 - larger and ruggedized, non-corrodable, spring-loaded contacts
 - low maintenance, adjustable mechanical system for probe mounting on different sizes of flanges
 - automated motion of the four point probe allowing flange scanning along circular pathways of different radii
3. Improvements in the electronic design of a low-resistance meter:
 - In order to be able to measure the resistance of large and thick flanges, the design of the presently used pulsed current resistance meter should be improved by: (i) providing higher amplitude current pulses (possibly up to 20-30 A); (ii) optimizing the pulse duration based on flange thickness and overheating limits; and (iii) automated application of repetitive current pulsing until satisfactory accuracy of resistance readings are reached.
 - A computerized data acquisition and processing system which will allow real time imaging of damage to a flange. Background resistances measured at locations where no crevices are present, will be subtracted from measured signals in order to increase the spatial and depth resolution of crevice recognition. Other techniques for increasing the resolution include: (i) comparison of resistance pattern measured with resistance images of non-corroded flanges of the same size and geometry stored in computer memory (pattern recognition); and (ii) correcting resistance data with theoretically predicted edge-effect correction factors.